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☐ 1. Document ID: JP 04054184 A

L1: Entry 1 of 2

File: JPAB

Feb 21, 1992

PUB-NO: JP404054184A

DOCUMENT-IDENTIFIER: JP 04054184 A

TITLE: 3-SUBSTITUTED CONDENSED TRIAZINE DERIVATIVE

PUBN-DATE: February 21, 1992

INVENTOR-INFORMATION:

NAME

COUNTRY

WATANABE, YOSHIFUMI

USUI, HIROYUKI

ASSIGNEE-INFORMATION:

NAME

COUNTRY

DAI ICHI SEIYAKU CO LTD

APPL-NO: JP02161842

APPL-DATE: June 20, 1990

INT-CL (IPC): C07D 471/04; A61K 31/425; A61K 31/53; A61K 31/53; A61K 31/535; A61K 31/55; C07D 487/04; C07D 487/04; C07D 498/04; C07D 498/04; C07D 498/04; C07D 513/04

ABSTRACT:

NEW MATERIAL: The 3-substituted condensed triazine derivative of formula I (X is hydroxy, halogen, alkylsulfonyloxy, trifluoromethanesulfonyloxy or arylsulfonyloxy; R is H, alkyl or aryl; m and n are 0-2; ring A is 5- or 7-membered ring which may contain one or two hereto atoms selected from N, O and S in addition to the nitrogen atom at the part condensed to triazine ring or may contain one or more double bonds).

EXAMPLE: 3-(2-Chloroethyl)-6,7,8,9-tetrahydro-2H-pyrido[1,2-a]-1,3,5-triazine-2,4(3H)-dione.

USE: A synthetic intermediate for the condensed triazine derivative of formula II which is an excellent serotonin 2 receptor antagonistic substance and effective for the prevention and remedy of circulatory diseases, etc.

PREPARATION: The compound of formula I is produced according to the reaction formula.

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Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KWIC
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☐ 2. Document ID: JP 04054184 A JP 3023691 B2

L1: Entry 2 of 2

File: DWPI

Feb 21, 1992

DERWENT-ACC-NO: 1992-110860
DERWENT-WEEK: 200019
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TITLE: 3-Substd. condensed tri:azine derivs. - for use as intermediates for drugs
inhibiting serotonin-2 receptor

PATENT-ASSIGNEE:

ASSIGNEE

DAIICHI PHARM CO LTD

CODE

DAUC

PRIORITY-DATA: 1990JP-0161842 (June 20, 1990)

PATENT-FAMILY:

PUB-NO	PUB-DATE	LANGUAGE	PAGES	MAIN-IPC
JP 04054184 A	February 21, 1992		013	
JP 3023691 B2	March 21, 2000		013	C07D471/04

APPLICATION-DATA:

PUB-NO	APPL-DATE	APPL-NO	DESCRIPTOR
JP 04054184A	June 20, 1990	1990JP-0161842	
JP 3023691B2	June 20, 1990	1990JP-0161842	
JP 3023691B2		JP 4054184	Previous Publ.

INT-CL (IPC): A61K 31/42; A61K 31/53; A61K 31/55; A61P 43/00; C07D 471/04; C07D 487/04; C07D 498/04; C07D 513/04

ABSTRACTED-PUB-NO: JP 04054184A
BASIC-ABSTRACT:

3-Substd. condensed triazine derivs. of formula (I) are new; where X = -OH, halogen atom, alkylsulphonyloxy, CF₃SO₂O-, or arylsulphonyloxy which may be substd. by one or more of halogen atom, alkoxy, alkyl and nitro; R = H, alkyl, or aryl which may be substd. by one or more of halogen atom, alkoxy, alkyl and trihalomethyl; m and n = integer of 0-2; the ring A = 5- or 7-membered ring which may contain 1 or 2 heteroatoms selected from N, O and S in addn. to the N atom at the condensed portion of the triazine ring and which may have one or more of double bonds.

(I) may be prepd. by reacting cpds. of formula (V) with ClCOOAr to give cpds. of formula (VI), reacting (VI) with a cpd. of formula (VII) to give (I) (X = -OH), and reacting (I) (X = -OH) with an alkylsulphonyl chloride, arylsulphonyl chloride or CF₃SO₂Cl to give (I) (X is not -OH).

USE - (I) are useful as intermediates for cpds. of formula (I) (X = gp. (III) or (IV); where R₁ = H, -OH, or aryl which may be substd. by 1 or more of halogen atom, -OH, alkyl, alkoxy and trihalomethyl; R₂, R₃ and R₄ = H, halogen atom, alkyl, alkoxy, or -CF₃; Ar₁ and Ar₂ = aryl or heteroaryl which may be substd. by one or more of halogen atom, -OH, alkyl, alkoxy and trihalomethyl) inhibiting serotonin-2 receptor which are useful in treatment of ischemic heart failure, cerebrovascular disturbance, depression, or schizophrenia.

In an example, to a soln. of 2.00g 6,7,8,9-tetrahydro-2H-pyrido(1,2-a)-1,3,5-triazine-2,4(3H)-dione, 1.13g 2-chloroethanol and 4.20 Ph₃P in 40 ml THF was dropwise added a soln. of 2.79g diethyl azodicarboxylate in 5 ml THF, and the mixt. was stirred for 1 hr. and evapd. in vacuo. The residue was chromatographed on a silica gel column (120g), eluted with MeOH/CHCl₃, and crystallised from acetone-i-Pr₂O to give 1.20g 3-(2-chloroethyl)-6,7,8,9-tetrahydro-2H-pyrido(1,2-a)-1,3, 5-triazine-2,4(3H)-dione. m.pt. 61-63 deg.C.

CHOSEN-DRAWING: Dwg.0/0

TITLE-TERMS: SUBSTITUTE CONDENSATION TRI AZINE DERIVATIVE INTERMEDIATE DRUG INHIBIT
SEROTONIN RECEPTOR

DERWENT-CLASS: B02

CPI-CODES: B06-H; B12-C06; B12-C10; B12-F01C; B12-F02; B12-G01;

CHEMICAL-CODES:

Chemical Indexing M2 *01*

Fragmentation Code

D014 D040 D860 E730 G001 G002 G010 G019 G040 G100
H2 H211 H341 H342 H343 H401 H481 H541 H542 H543
H600 H602 H603 H608 H609 H641 H642 H643 H681 H682
H685 H686 J5 J522 K432 L640 L9 L910 M210 M211
M212 M213 M214 M215 M216 M220 M221 M222 M231 M232
M233 M240 M271 M272 M280 M281 M282 M283 M311 M312
M313 M314 M315 M321 M322 M323 M331 M332 M333 M334
M340 M342 M343 M344 M352 M353 M362 M373 M383 M391
M392 M393 M412 M511 M520 M530 M531 M532 M540 M710
M903 M904

Ring Index

00930 47069 59142 59431

Markush Compounds

199214-22301-N

SECONDARY-ACC-NO:

CPI Secondary Accession Numbers: C1992-051444

Full	Title	Citation	Front	Review	Classification	Date	Reference	Sequences	Attachments	Claims	KMC
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⑫ 実用新案公報(Y2)

平4-54184

⑮ Int. Cl.⁵

識別記号

庁内整理番号

②④ 公告 平成4年(1992)12月18日

C 01 B 5/02

Z

9041-4G

(全3頁)

⑭ 考案の名称 $H_2^{15}O$ 合成用反応装置

⑯ 実 願 昭61-179587

⑰ 公 開 昭63-85630

⑱ 出 願 昭61(1986)11月20日

⑲ 昭63(1988)6月4日

⑳ 考 案 者 西 原 善 明 愛媛県新居浜市惣開町5番2号 住友重機械工業株式会社
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㉒ 復 代 理 人 弁理士 佐田 守雄

審 査 官 寺 本 光 生

㉓ 参 考 文 献 特開 昭60-4900(JP, A) 特公 昭48-20104(JP, B1)

1

2

㉔ 実用新案登録請求の範囲

1 電気抵抗の立ち上がり温度が約150~200℃の間にある無機質半導体物質粉末を多数の透孔を有する成形体に成形し、この透孔の内面に白金もしくはパラジウムの如き酸化触媒を担持させ、かつ成形体の半導体物質内には加熱用導線を取りつけて反応容器内に収容したことを特徴とする $H_2^{15}O$ 合成用反応装置。

2 無機半導体物質がチタン酸バリウムである実用新案登録請求の範囲第1項に記載の装置。

考案の詳細な説明

〔技術分野〕

本考案は酸素の同位体元素である $^{15}O_2$ と水素とを反応させて H_2^{15} を製造するための装置に関する。

〔公知技術〕

半減期の短い放射性同位元素を成分として含む化合物は各種のマーキング剤や検査剤として種々利用されるようになってきている。殊に小型のサイクロトロン の出現により放射性同位元素が得易くなつてからはその元素の化合物を使用直前に製造することが可能となつたので、その利用範囲は急速に拡大しつつある。本出願前に本発明者の発明にかゝる「放射性同位液体合成用反応器」が特開昭60-4900号(特願昭58-11902号)として公知と

なっている。この特開昭60-4900号公報には放射性化合物の製造装置が具体的に図示説明されている。

〔本考案で解決しようとする問題点〕

5 放射性同位元素中 $^{15}O_2$ は半減期が約2分と極めて短く H_2^{15} の合成も他の放射性同位元素よりもより早く合成しなければならない。従つて前記公開公報に記載の装置も使用し得るが、より反応が完全でより安全に反応を進行させる装置が望まし

10 い。

〔問題点の解決策〕

本考案は上記の問題点を使用触媒を特定することにより解決したものであり、より具体的には触媒の担体に電気抵抗の立ち上がり温度が約150~200
15 °C程度の無機質半導体物質粉末を多孔体として成形し、この担体上に白金あるいはパラジウムを酸化触媒として担持させ、この成形された半導体物質内に加熱用導線を取りつけ通電することにより、合成反応を自動的に調節された温度で行い得るようにしてこの問題を解決したものである。

〔発明の構成〕

即ち本考案は、電気抵抗の立ち上がり温度が約150~200℃の間にある無機質半導体物質粉末を多数の透孔を有する成形体に成形し、この透孔の内面
25 に白金もしくはパラジウムの如き酸化触媒を担持

させ、かつ成形体の半導体物質内と前記触媒面とは夫々加熱用導線を取りつけて反応容器内に収容したことを特徴とする $H_2^{15}O$ 合成用反応装置を要旨としており、半導体物質としてはチタン酸バリウムが好ましい。

〔構成の説明〕

以下本考案を図に基づいて説明する。第1図は本考案装置の側断面図であり、本考案の要部以外の部分も図示している。また第2図は本考案装置に用いられる成形触媒の平面図であり、成形した触媒と加熱用導線との関係位置を示すものである。第1図で示された本考案の装置は前述した特開昭60-4900号公報に記載の図面の第1図、第2図A~Cと一見近似しているが、外部加熱手段がないこと及び特定された構造の自己加熱性触媒を用いていることで前記公報に記載された装置とは本質的に相違している。

さて第1図において1は反応容器であり、その底部には流体は透過する触媒止め2が設けられ触媒は反応容器外に出ないようになっている。3は反応器で生成した $H_2^{15}O$ を凝縮露化させるための漏斗状容器、4はそれに続く蛇管を示している。また5は漏斗状容器に水あるいはバージ用不活性ガスを注入して3の漏斗状容器壁に凝縮した $H_2^{15}O$ を含む水を流去するための導管である。

また第1図および第2図において6は触媒成形体を示し、この触媒成形体には孔の内面に触媒金属が担持されている気体透過性孔、即ち触媒反応孔6'が多数触媒の軸方向に設けられている。更に触媒成形体には一対の加熱用導線7, 7'が取り付けられており、また半導体物質に電圧を与えることができる様になっている(但し電源は示されていない)。

さて承知のように水素と酸素はその発火点以上では急激な発熱反応を起こすが、発火点は以外に高く550°Cにも達する。従つて酸素及び水素を反応させるためには、酸素や水素濃度が極めて低い場合であつてもその発火点に達する程の加熱手段や補助的な触媒も使用する必要がある。

本考案では特別の加熱装置を使用することなく反応温度を自動的に制御する装置を提供するもの

である。本考案においては触媒として白金あるいはパラジウムを使用する。この両金属は100°Cを越え150°C付近において酸化触媒としての機能を急激に発揮し初めるが、余り高い温度では触媒被毒が生ずるので好ましくはない。そのため本考案では触媒の加熱制御を担体物質の電気抵抗の立ち上り温度が約150~200°Cのものを成形しこの問題を解決することに成功したものである。現在のところ最も好ましい物質としてはチタン酸バリウムであるが、無機質の混合物などで同様の効果を奏し得るものがある程度想定され得るので、本考案の触媒担体はチタン酸バリウム(説明はこれについて行うが)に限られるものではない。

チタン酸バリウムの電気抵抗の立上りは170~180°Cにある。従つてこれを成形し定圧の電流を通すると、この成形物は170~180°Cで熱の発生は止まり定温となり温度低下が生ずれば又電流が通じて、ほぼ成形体は170°C付近に保たれる。

従つて不活性キャリアーガスで稀められた $^{15}O_2$ を含む酸素含有ガス及び同じく不活性キャリアーガスで稀められた H_2 ガスが管8及び9から反応器1内の触媒反応孔内へ送られると直ちに反応し $H_2^{15}O$ を含んだ水が生成する。この反応は発熱反応であるが、酸素と水素の量が少ないのでその熱はガス体である反応生成物の昇温に消費されるので過熱の恐れがなく、過熱されれば触媒成形体は発熱を停止するので、触媒反応孔内の温度を100~150°Cの間に常に保つことができ温度制御は自動的に行なわれることになる。しかも反応温度が低いので触媒の被毒は生じない。

〔考案の効果〕

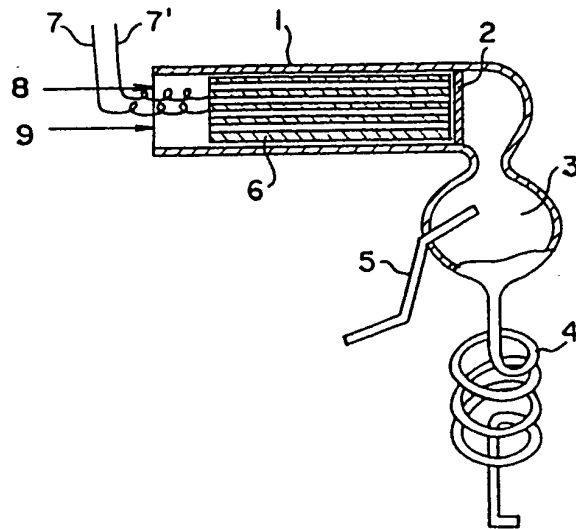
本考案によればこの種装置に必要な加熱手段が不要になるので装置は簡単となり、又反応の温度制御が自動となる利点がある。

35 図面の簡単な説明

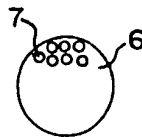
第1図は本考案の側断面説明図であり、第2図は触媒成形体の平面図である。

1……反応容器、2……触媒止め、3……漏斗状容器、4……蛇管、5……導管、6……触媒成形体、6'……触媒反応孔、7, 7'……加熱用導線、8……酸素供給管、9……水素供給管。

第 1 図



第 2 図



PTO 03-1245

Japanese Utility Model Application No.
Hei 4[1992]-54184

REACTION APPARATUS FOR SYNTHESIZING H_2^{15}O

Yoshiaki Nishihara

UNITED STATES PATENT AND TRADEMARK OFFICE
WASHINGTON, D.C. JANUARY 2003
TRANSLATED BY THE RALPH MCELROY TRANSLATION COMPANY

JAPANESE PATENT OFFICE
PATENT JOURNAL
UTILITY MODEL APPLICATION NO. HEI 4[1992]-54184

Int. Cl. ⁵ :	C 01 B 5/02
Sequence Nos. for Office Use:	Z 9041-4G
Filing No.:	Sho 61[1986]-179587
Filing Date:	November 20, 1986
Kokai No.:	Sho 63[1988]-85630
Kokai Date:	June 4, 1988
Publication Date:	December 18, 1992
	(Total of 3 pages)
Examination Request:	Not filed

REACTION APPARATUS FOR SYNTHESIZING H_2^{15}O

[H_2^{15}O gosei yo hannosochi]

Inventor:	Yoshiaki Nishihara
Applicant:	Sumitomo Heavy Industries, Inc.

[There are no amendments to this patent.]

Claims

1. A reaction apparatus for synthesizing H_2^{15}O characterized by the following facts: an inorganic semiconductor substance powder with an electric resistance rising temperature in the range of about 150-200°C is molded into a molded body having many through holes; an oxidation catalyst, such as platinum or palladium, is carried on the inner surface of the through holes; wires used for heating are installed in the semiconductor substance of the molded body, which is accommodated in a reaction container.

2. The apparatus described in Claim 1 characterized by the fact that the inorganic semiconductor substance is barium titanate.

Detailed explanation of the design

The present design pertains to an apparatus used for manufacturing H_2^{15}O from a reaction between hydrogen and $^{15}\text{O}_2$, an isotope of oxygen.

Prior art

Compounds containing radioactive isotopes with short half-life are used as various types of marking agents or inspecting agents. In particular, since radioactive isotopes can be obtained easily due to the emergence of small-scale cyclotron, the compounds of these elements can be manufactured right before use. The application range of such compounds has been expanded quickly. As another invention by the present inventor, "Reactor for synthesizing radioactive isotopic liquid" was publicized as Japanese Kokai Patent Application No. Sho 60[1985]-4900 (Japanese Patent Application No. 58[1983]-11902) prior to the present patent application. The radioactive substance manufacturing apparatus disclosed in Japanese Kokai Patent Application No. Sho 60[1985]-4900 will be explained in detail with reference to figures.

Problems to be solved by this design

Among the radioactive isotopes, $^{15}\text{O}_2$ has a half-life as short as about 2 min. Therefore, H_2^{15}O must be synthesized earlier than other radioactive isotopes. Consequently, although the apparatus disclosed in the aforementioned patent can be used, it is desired to develop an apparatus that can carry out the reaction more safely.

Means for solving the problems

The present design solves the aforementioned problem by specifying the catalyst used. More specially, an inorganic semiconductor substance with an electric resistance rising temperature in the range of about 150-200°C is molded into a porous body used as the catalyst carrier. Platinum or palladium is carried as catalyst on the carrier. Wires used for heating are installed in the molded semiconductor substance. When the power is turned on, the synthesizing reaction can be carried out at an automatically adjusted temperature. In this way, the aforementioned problem is solved.

Configuration of the invention

In other words, the present design provides a reaction apparatus for synthesizing H_2^{15}O characterized by the following facts: an inorganic semiconductor substance powder with an

electric resistance rising temperature in the range of about 150-200°C is molded into a molded body having many through holes; an oxidation catalyst, such as platinum or palladium, is carried on the inner surface of the through holes; wires used for heating are installed in the semiconductor substance of the molded body, which is accommodated in a reaction container. The inorganic semiconductor substance is preferably barium titanate.

Explanation of the configuration

In the following, the present design will be explained based on figures. Figure 1 is a side cross-sectional view illustrating the apparatus disclosed in the present invention. Other parts besides the main parts of the present design are also shown. Figure 2 is a plan view illustrating the molded catalyst used in the apparatus of the present design. Although the apparatus of the present design shown in Figure 1 looks similar to the apparatus shown in Figures 1, 2(A)-(C) in said Japanese Kokai Patent Application No. Sho 60[1985]-4900, it is virtually different from the apparatus described in the aforementioned patent application in the fact that there is no external heating means and the fact that a self-heating catalyst with a specified structure is used.

In Figure 1, (1) represents a reaction container. A catalyst stopper (2) that allows fluid to pass through is installed at the bottom of the reaction container to prevent the catalyst from flowing out of the reaction container. (3) represents a hopper-shaped container where the H_2^{15}O generated in the reactor is condensed. (4) represents a coil pipe connected to the hopper-shaped container. (5) represents a guiding pipe, which injects water or inactive gas for purging into the hopper-shaped container to flow away the water containing the H_2^{15}O condensed on the wall of hopper-shaped container (3).

Also, in Figures 1 and 2, (6) represents a molded catalyst body. Many gas permeable holes, that is, catalyst reaction holes (6') with a catalytic metal carried on the inner surface of the holes are formed in the molded catalyst body in the axial direction of the catalyst. Also, a pair of wires (7), (7') used for heating are installed in the molded catalyst body, and a voltage can be applied to the semiconductor substance (however, the power supply is not shown).

It is a well-known fact that hydrogen and oxygen will have an intense exothermic reaction above the ignition point. The ignition point, however, is as high as 550°C. Consequently, in order to make hydrogen and oxygen react with each other, even if the concentrations of oxygen and hydrogen are extremely low, it is necessary to use a heating means that can raise the temperature to the ignition point as well as an auxiliary catalyst.

The present design provides an apparatus that uses no special heating device and is able to control the reaction temperature automatically. In the present design, platinum or palladium is used as the catalyst. At the beginning, the two metals can display sharp function as an oxidation catalyst when the temperature exceeds 100°C and approaches 150°C. However, if the

temperature is further raised, catalyst poisoning will occur. Therefore, in the present design, the problem related to the control over catalyst heating is solved successfully by molding a carrier substance with an electric resistance rising temperature in the range of about 150-200°C.

Currently, the most preferable substance is barium titanate. However, since the same effect may also be realized using a mixture of inorganic substances, the catalyst carrier used in the present design is not limited to barium titanate (although it is used in the explanation).

The electric resistance rising temperature of barium titanate is in the range of 170-180°C. Consequently, when this material is molded and a current is supplied at a constant voltage, heat generation in the molded body is stopped at 170-180°C, and the temperature is kept constant. If the temperature drops, the current is supplied again to keep the temperature of the molded body near 170°C.

Consequently, when an oxygen-containing gas containing $^{15}\text{O}_2$ diluted with an inactive carrier gas and H_2 gas diluted with the same inactive carrier gas are fed from pipes (8) and (9) into the catalyst reaction hole in reactor (1), they react with each other immediately to generate water containing H_2^{15}O . Although this reaction is an exothermic reaction, since the amounts of the oxygen and hydrogen are very small, the heat is consumed during heating of the reaction product. Therefore, there is no need to worry about overheating. Since the molded catalyst body stops heat generation if it is overheated, the temperature in the catalyst reaction holes can be constantly kept in the range of 100-150°C. The temperature can be controlled automatically. Also, since the reaction temperature is low, no catalyst poisoning will occur.

Effect of the design

According to the present design, since no special heating device is needed for this type of apparatus, the apparatus can be simplified. Also, the reaction temperature can be controlled automatically.

Brief description of figures

Figure 1 is a side cross-sectional view illustrating the present design. Figure 2 is a plan view illustrating the molded catalyst body.

- 1 Reaction container
- 2 Catalyst stopper
- 3 Hopper-shaped container
- 4 Coil pipe
- 5 Guiding pipe
- 6 Molded catalyst body

- 6' Catalyst reaction hole
7, 7' Wires for heating
8 Oxygen supply pipe
9 Hydrogen supply pipe

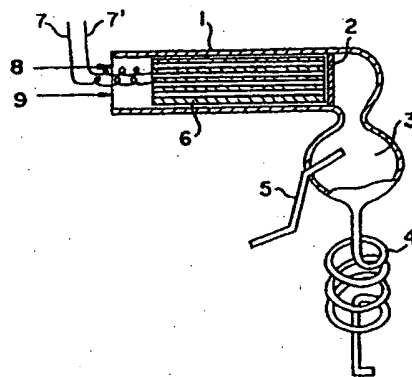


Figure 1

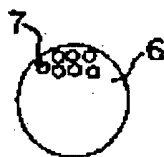


Figure 2